IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No.	: 10/435,008	Confirmation No.:	3596
Applicant(s)	: Dang et al.		
Filed	: January 12, 2004		
TC/AU	: 1711		
Examiner	: Duc Truong		
Docket No.	: AFD 645		
Customer No.:	26902		

Commissioner for Patents
P.O. Box 1450
Alexandria VA 22313-1450

DECLARATION OF NARAYANAN VENKATASUBRAMANIAN
Under Rule 1.131

1. I, Narayanan Venkatasubramanian, declare that:

2. I have a B.S. in Chemistry from the University of Madras, India, an M.S. in Chemistry from the Indian Institute of Technology, Madras, India and a PhD in Chemistry from the Indian Institute of Technology, Madras, India.

3. I have been working in research and development in the Polymer Chemistry/Polymeric Materials art at the University of Dayton Research Institute, Dayton, OH since December 1999.

4. I am one of the inventors of the subject matter of the above-identified application.

5. The following facts show a conception and reduction to practice of the above-identified invention before April 7, 2002:

Before April 7, 2002, I developed new rigid-rod benzobisazole polymer compositions incorporating 1,5-naphthalene-diyl units for potential utilization as non-conducting high modulus fibers in structural composites for Air Force applications. Besides conventional reinforcement, other areas of application for these high performance polymers include protective garments, ballistic vests and abrasion- and flame-resistant fabrics. The invention takes advantage of the unique conformational possibilities and torsional behavior of the polybenzobisazole chains containing 1,5-naphthalic segments which will influence the mechanical properties of the polymeric fibers. This development is described in University of Dayton Research Institute (hereinafter referred to as "UDRI") Technology Disclosure No.349. A copy of this Technology Disclosure Form is attached.

In particular, the Page 3 of 5 of the attached Technology Disclosure Form show that the relevant dates are as follows:

1,5-Naphthalenedicarboxylic acid (monomer) (07/23/01):

A new method is described for the preparation of this diacid monomer starting with 1,5-diaminonaphthalene as starting material. The process of conversion of 1,5-naphthalenedinitrile to the dicarboxylic acid monomer is described with the date of 07/23/01.

1,5-Naphthalenebenzobisthiazole (polymer) (08/28/01 and again, 11/27/01):

The high temperature polycondensation of 1,5-naphthalenedicarboxylic acid with 2.5-diamino-1,4-benzenedithiol dihydrochloride in polyphosphoric acid with final polymer concentrations of 10 wt % and 12 wt % respectively are described on the dates as indicated above.

1,5-Naphthalenebenzobisoxazole (polymer) (02/15/02):

The high temperature polycondensation of 1,5-naphthalenedicarboxylic acid with 4,6-diamino-1,3-benzenediol dihydrochloride in polyphosphoric acid , with a final polymer concentration of 14 wt %, is described on the date indicated above.

6. All acts described herein were conducted in the United States of America before April 7, 2002.

7 I further declare that all statements made herein are of my own knowledge and are true, and that all statements made on information and belief are believed to be true; and further that the statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001, Title 18, of the United States Code and that such willful false statements may jeopardize the validity of the above-identified application or any patent issue thereon.

Dated: 08/12/2005

Narayanan Venkatasubramanian

AFD 645 Rule 131.doc

Disclosure No. 349
Log-In-Date 2/24/03
OFFICE USE ONLY

UNIVERSITY OF DAYTON
TECHNOLOGY DISCLOSURE FORM - INVENTIONS

1. Descriptive Title of Invention

Rigid-rod Benzobisazole Polymers Incorporating Naphthalene-1,5-diyl Structural Units

2. Inventor(s)

Name (typed) (1) Thuy D. Dang

Signature Thuy Dinh Dang

Date 12/11/02

Employer Polymer Branch, AFRL/MLBP, Wright-Patterson AFB, OH 45433

Business Phone No. 937-255-0042

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Signature N. Venkatasubramanian

Date 12/11/02

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Business Phone No. 937-255-9117

Home Address 2582 King Arthur Drive Beavercreek OH 45431

Name (typed) (3) Dr. Jar-Wha Lee

Signature Jar Wha Lee

Date 12/13/02

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JWLEESTI@worldnet.att.net

Business Phone No. 614-850-7314

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Name (typed) (4) Dr. Soo-Young Park

Signature

Date

Employer (Current) .
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Kyungpook National University
#1370 Sangyuk-dong, Buk-Gu, Daegu 702-701, Korea

Business Phone No. 82-53-950-5630

Home Address 107-1708, WhaSung 3 Cha, Dongchun-dong, Buk-Gu, Daegu 702-797, Korea

Disclosure No. _____

Log-In-Date _____

OFFICE USE ONLY

UNIVERSITY OF DAYTON

TECHNOLOGY DISCLOSURE FORM - INVENTIONS

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2. Inventor(s)

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Date 12/18/2002

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3. Witnesses: The disclosure shall be signed by two witnesses who are not inventors of any part of this invention.

Name (typed) Dr. Jong-Beom Baek

Signature

Date

Name (typed) Dr. Balasubramanian Sankaran

Signature

Date

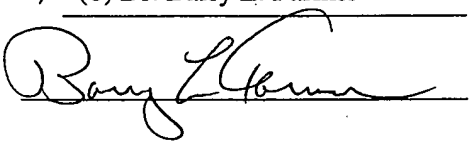
Disclosure No._____
Log-In-Date _____
OFFICE USE ONLY

UNIVERSITY OF DAYTON
TECHNOLOGY DISCLOSURE FORM - INVENTIONS

1. Descriptive Title of Invention

   Rigid-rod Benzobisazole Polymers Incorporating Naphthalene-1,5-diyl Structural Units

2. Inventor(s)

Name (typed) (5) Dr. Fred E. Arnold	Name (typed) (6) Dr. Barry L. Farmer
Signature	Signature 
Date	Date
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Home Address 1583 Ambridge Road Centerville OH 45459	Home Address 1522 Kathy Marie Ct., Xenia, OH 45385
Name (typed)	Name (typed)
Signature	Signature
Date	Date
Employer	Employer
Business Phone No.	Business Phone No.
Home Address	Home Address

3. Witnesses: The disclosure shall be signed by two witnesses who are not inventors of any part of this invention.

Name (typed) Dr. Jong-Beom Baek	Name (typed) Dr. Balasubramanian Sankaran
Signature	Signature
Date	Date

Disclosure No._____
Log-In-Date _____
OFFICE USE ONLY

UNIVERSITY OF DAYTON
TECHNOLOGY DISCLOSURE FORM - INVENTIONS

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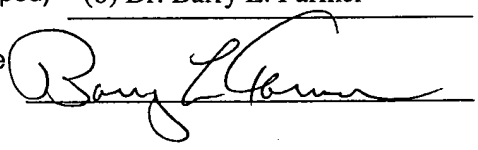
Signature _____	Signature 
Date _____	Date _____
Employer Polymer Branch, AFRL/MLBP, Wright-Patterson AFB, OH 45433	Employer Materials Directorate, AFRL/ML, Wright-Patterson AFB, Dayton, OH 45433
Business Phone No. _____	Business Phone No. 937-255-6825
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Name (typed) _____	Name (typed) _____
Signature _____	Signature _____
Date _____	Date _____
Employer _____	Employer _____
Business Phone No. _____	Business Phone No. _____
Home Address _____	Home Address _____

3. Witnesses: The disclosure shall be signed by two witnesses who are not inventors of any part of this invention.

Name (typed) Dr. Jong-Beom Baek	Name (typed) Dr. Balasubramanian Sankaran
Signature _____	Signature _____
Date	Date

3. Witnesses: The disclosure shall be signed by two witnesses who are not inventors of any part of this invention.

Name (typed) Dr. Jong-Beom Baek	Name (typed) Dr. Balasubramanian Sankaran
Signature Baek Jongbeom	Signature
Date 12/27/02	Date 12/23/02

4. Approval: The disclosure shall be signed by the Research Institute division head and/or the academic department chair as applicable.

Name (typed) CHYI-SHAN WANG Name (typed) ALLAN S. CRASTO

Signature [signature] Signature [signature]

Date 01/14/03 Date 1-17-03

5. Was the work leading to the invention performed (in whole or in part) on an externally sponsored program? ☐ Yes ☐ No

Sponsoring Agency/Firm AFOSR and Wright-patterson AFB

Account Number 2423010362 Contract Number F33615-00-D-5008

6. Date and circumstances of first verifiable record of the invention:

The synthesis of the monomer, i.e., 1,5-naphthalenedicarboxylic acid by the hydrolysis of the corresponding dinitrile was reported on 07/23/01. The synthesis of the polybenzobisthiazole composition incorporating naphthalene-1,5-diyl structural unit was first reported on 08/28/01.

7. Date and description of other written records of the invention predating this disclosure:

The preparation of the polybenzobisthiazole composition incorporating naphthalene-1,5-diyl unit was again reported on 11/27/01, varying the polymer concentration in the polyphosphoric acid medium. The synthesis of the corresponding polybenzobisoxazole composition was reported on 02/15/02.

8. Is there a laboratory notebook record of this invention? ☒ Yes ☐ No

Notebook number or other identification UDRI # 768 and # 789

Page numbers # 768 (page #s 76, 83), # 789 (page #s 1 and 20).

9. Has the invention been demonstrated experimentally? ☒ Yes ☐ No

Date 08/28/01 and 02/15/02 Where and How (explain below):

The compositions were synthesized in high molecular weights as evidenced by their intrinsic viscosities and displayed the lyotropic liquid crystallinity characteristic of rigid-rod polymers. Both the polybenzobisthiazole and the polybenzobisoxazole compositions were continuously spun into fibers from the anisotropic polyphosphoric acid dopes by a dry jet-wet spinning method with draw ratios ranging from 20-35 by one of the co-inventors, Dr. Jar Wha Lee.

10. First Public Disclosure. Identify the names, places, and dates associated with the first disclosure of pertinent details of the invention to anyone outside the University without the benefit of a formal confidentiality agreement. Public disclosure may be made in the following ways: (1) an oral presentation to a scientific meeting or an informal group;(2) circulation of an abstract of a talk; (3) publication of a journal article or news story; (4) delivery and distribution of a contract report, etc. Attach copies of any publications. If you are not sure whether public disclosure has been made, give the details of all external communication concerning the invention. If there has been no outside disclosure, so indicate.

    The first public disclosure appeared as "Polymer Preprints" published by the Polymer Division, American Chemical Society. The papers were presented at the annual Spring ACS Meeting at Orlando, Florida, April 7-11, 2002; the citations are given below.

    1. " Synthesis and Characterization of Rigid-rod Benzobisazole Polymers Incorporating Naphthalene-2,6- and 1,5-diyl Structural Units", Thuy D. Dang, N. Venkatasubramanian, Adam Talicska, Soo-Young Park and Fred E. Arnold, Polymer Preprints (ACS), 2002, 43(1), 660-661

    2. " Structural Studies on Naphthalene-based Rigid-rod Benzobisthiazole Polymers", Soo-Young Park, Jarwha Lee, N. Venkatasubramanian, Thuy D. Dang, Fred E. Arnold and B. L. Farmer, Polymer Preprints, 2002, 43(1), 248-249

11. Sale of Product. Has a purchase order been accepted for sale of the result of the invention in any form?
    ☐ Yes ☒ No

    If YES, please provide pertinent details

12. Samples. Have samples of the invention been given to anyone outside the University for evaluation (including sponsors)? ☒ Yes ☐ No

    Please provide pertinent details:

    Dr. Soo-Young Park, our research collaborator and faculty member in the Dept. of Polymer Science, Kyungpook National University is currently evaluating the mechanical properties of as spun and heat treated fibers including their compressive modulus and strength.

13. What do you see as the commercial value of the invention? What is the market and how large is it?

    The commercial value of the inventions stems from their potential for utilization as reinforcing, non-conducting high tenacity polymeric fibers in structural composites. The scope for commercial utility of these fibers is dependent on significant improvement in compressive strength relative to the state of-art rigid-rod polymeric fibers such as PBO.

14. List the names of firms that might be interested in licensing the invention.

15. Attach to this form a Nonconfidential Abstract of the invention addressing the items listed below:

(a) Description. Provide a brief general description that communicates the essence of the invention without disclosing pertinent technical details.

(b) Application. Intended use of the invention, especially for commercial purposes. Be specific.

(c) Advantages. What is new and useful about the invention? Why is it better than the prior art?

(d) Current State of Development. Is the invention a concept only, fully developed and ready to license, or somewhere in between? Give some idea of how much development work would be needed to commercialize the invention.

16. Attach to this form a Detailed Description of the invention according to the following instructions. Type the description space-and-a-half, use as many pages as necessary, and number the pages consecutively. Prepare the Detailed Description so an individual reasonably skilled in the art would readily recognize what is new, different, useful, and non-obvious about the invention. Try to communicate the central essence of the invention. Use photographs, sketches, and graphs as necessary. Include the following elements in the description:

(a) Intent. Briefly identify the problem and/or need addressed by the invention.

(b) Applicability. Describe the invention's general areas of application and specific uses.

(c) Function. Describe in detail the pertinent features of the invention with emphasis on (1) novelty, (2) advantages, (3) disadvantages and limitations, and (4) prior related inventions.

(d) Inventors' Roles. If more than one inventor contributed to the invention, describe the individual roles and contributions of each inventor to either the concept or its successful reduction to practice.

Submit completed disclosure form to:

THE UNIVERSITY OF DAYTON
Research Institute
Technology Commercialization Office
KL 503
300 College Park
Dayton, OH 45469-0102
Telephone 937-229-3515
Fax 937-229-3433

POLYMER PREPRINTS

Volume 43 *Number 1* *Spring 2002*

Published by the Division of Polymer Chemistry, Inc.

American Chemical Society

Papers presented at the Orlando, Florida Meeting

223rd ACS NATIONAL MEETING
Orlando, Florida
April 7-11, 2002

SELF-ASSEMBLED PHOTONIC BAND GAP MATERIALS

(N. P. Balsara and E. L. Thomas, Organizers)

TUTORIAL

Sunday Morning

SESSION I

Sunday Afternoon

SYNTHESIS AND CHARACTERIZATION OF RIGID-ROD BENZOBISAZOLE POLYMERS INCORPORATING NAPHTHALENE 2,6- AND 1,5-DIYL STRUCTURAL UNITS

Thuy D. Dang[1], N. Venkatasubramanian[2], Adam Talicska[3], Soo-Young Park[1] and Fred E. Arnold[1]

1. AFRL/MLBP, Materials & Manufacturing Technology Directorate, Wright-Patterson Air Force Base, 2941, P Street, suite 1, Dayton, OH 45433-7750
2. University of Dayton Research Institute, 300 College Park Drive, Dayton, OH, 45469
3. Department of Biology, Wright State University, Dayton, OH 45435

Introduction

Aromatic heterocyclic rigid-rod polymers such as poly(p-phenylenebenzobisthiazole)[1] (PBT) and poly(p-phenylenebenzobisoxazole)[2] (PBO) are well-known for their unique mechanical properties and exceptional thermal and thermooxidative stabilities. Among this class of materials, PBO fibers and yarns have been commercialized under the trademark of 'Zylon' by Toyobo Industries and have tensile properties superior to those of Kevlar[2]. The myriad applications of PBO range from conventional reinforcement to protective garments, ballistic vests as well as abrasion- and flame-resistant fabrics, to name just a few. This paper is concerned with the synthesis and characterization of rigid-chain polybenzobisazoles incorporating naphthalene-2,6- and 1,5-diyl moieties in the backbone.

A literature search revealed the presence of a single patented report[3] dealing with a copolymer involving PBO and a polybenzobisoxazole linked to the naphthalene-2,6-diyl unit. The rationale for the current research was to take up a systematic investigation of the naphthalene-based polybenzobisazole polymers. The extended planar packing afforded by the naphthalene-2,6-diyl[4] and 1,5-diyl units in the polybenzobisazole is an interesting additional molecular feature from the standpoint of consideration of extended-chain conformations as well as the torsional behavior relative to PBT and PBO. The planar zig-zag conformation of the polymer chain in which the alternating benzobisthiazole units have a cis-disposition relative to each other and a polymer chain conformation in which the heterocyclic units have an all-trans disposition, are depicted below (**Figure 1**) for the 1,5- naphthalene-PBT system.

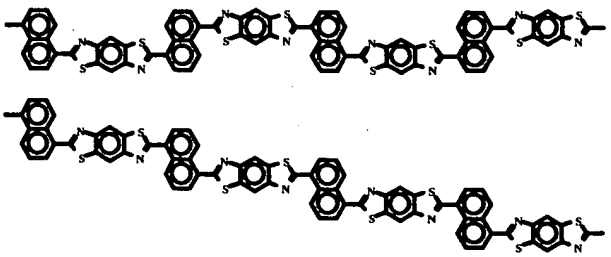


Figure 1. Possible polymer chain conformations in the polybenzobisthiazole incorporating Naphthalene-1,5-diyl structural unit

Detailed analyses of the chain packing and the chain conformations in the polybenzobisthiazole system incorporating the isomeric naphthalene-diyl units based on X-ray diffraction studies of the polymeric fibers and on molecular modeling, form the subject of a companion paper[5]. Another potential application of interest will be the study of the influence of polymer chain packing on the compressive properties of these organic fibers.

Experimental

Materials. 2,6-Naphthalenedicarboxylic acid was received from TCI America and used without further purification. 1,4-Diamino-2,5-benzenedithiol dihydrochloride and 4,6-diamino-1,3-benzenediol dihydrochloride were obtained through custom synthesis by known preparative routes. They were further purified by recrystallization in the presence of $SnCl_2.2H_2O$ and Conc.HCl prior to polymerization.

Synthesis of 1,5-naphthalenedicarboxylic acid. The monomer was synthesized from 1,5-diaminonaphthalene via the reaction of its diazonium salt with KI to form 1,5-diiodonaphthalene followed by the conversion to 1,5-naphthalenedinitrile which was hydrolyzed by HBr/AcOH to form the desired dicarboxylic acid in satisfactory yields.

Synthesis of polybenzobisthiazole based on naphthalene-1,5-diyl unit (1,5-NaphPBT). 1,5-Naphthalenedicarboxylic acid (2.5 g, 0.0116 mole) was reacted with equimolar amounts of 1,4-diamino-2,5-benzenedithiol dihydrochloride (DABDT, 2.8352 g) in polyphosphoric acid (PPA) with 77 % initial concentration of P_2O_5 . After the completion of dehydrochlorination of DABDT at 105^{0}C, P_2O_5 was added to "adjust" the final PPA composition to have 83 % P_2O_5 content[6]. The final polymerization temperature was maintained at 180^{0}C for a day. The viscous polymer product (10 wt % in PPA) was found to be stir-opalescent. The polymer was precipitated in water, broken up in a blender, soxhlet-extracted with water and finally dried for 48 hours *in vacuo* at 100^{0}C to obtain a fibrous yellow solid.

Results and Discussion

The preparative scheme for the synthesis of 1,5-naphthalenedicarboxylic acid monomer is described below in **Figure 2**. The monomer was purified via dissolution in aqueous $NaHCO_3$ as the sodium salt and reprecipitation by acidification.

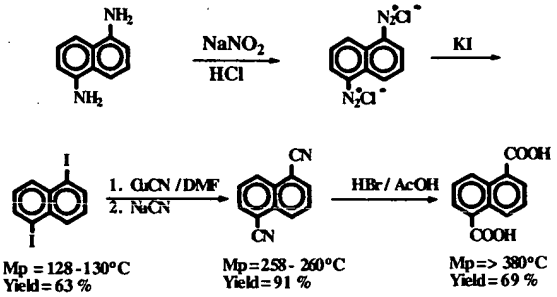


Figure 2. Synthesis of 1,5-Naphthalenedicarboxylic acid

The polycondensation reactions to synthesize the polybenzobisthiazoles based on naphthalene-2,6- and 1,5-diyl structural units (2,6-NaphPBT and 1,5-NaphPBT) and polybenzobisoxazole incorporating naphthalene-2,6-diyl unit (2,6-NaphPBO) are described in **Figure 3**.

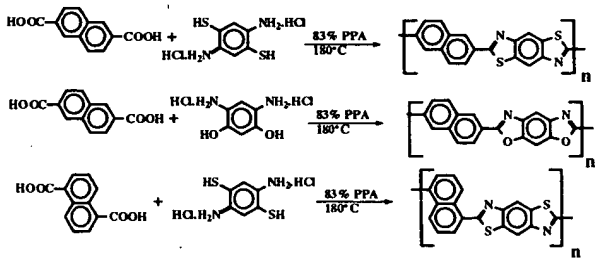
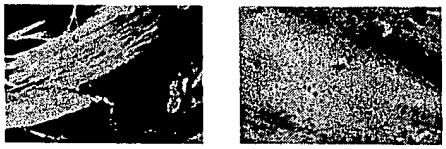


Figure 3. Synthesis of polybenzobisazoles incorporating Naphthalene-2,6- and 1,5- diyl structural units

The polymerization reactions were conducted in relatively high concentrations (10-12 wt % polymer in PPA). The viscous, dark purple dopes exhibited stir-opalescence characteristic of the lyotropic liquid crystallinity of the rigid-rods in the 175-180^0 C range in which the polycondensation took place. Further evidence for the presence of the ordered phase was obtained by the observation of optical birefringence of the sample sealed between glass slides and examined under polarizing optical microscopy. The anisotropic texture was found to be persistent in these polymer systems, even several days after the initial sample preparation. The optical micrographs of both polybenzobisthiazoles described in the current study are shown in **Figure 4.**

The polymers were also amenable to being continuously spun into fibers from the nematic phase, as described elsewhere[5].



(A) (B)

Figure 4. Optical birefringence of 10 wt % rigid-rod polymer in PPA (A) 2,6-NaphPBT (B) 1,5-NaphPBT

2,6-NaphPBT and 2,6-NaphPBO were isolated as fibrous, purple solids and 1,5-NaphPBT was obtained as a fibrous, dark yellow solid. The polymers could be solubilized only in strong protic acids such as methanesulfonic acid (MSA). The solution properties of the polybenzobisazoles are listed below in **Table 1.**

Table 1. Solution properties of Polybenzobisazoles incorporating Naphthalene-2,6- and 1,5-diyl units

Benzobisazole polymer	Polymerization conditions (wt % polymer in PPA)	Intrinsic viscosity (η) in dl/g (MSA, 30°C)
2,6-NaphPBT	12	41.0
2,6-NaphPBT	10	27.0
2,6-NaphPBO	10	21.0
1,5-NaphPBT	10	13.2

The intrinsic viscosities obtained for these polymers for an initial concentration of 0.10 g/dl in MSA, were indicative of their high molecular weights. Particularly remarkable was the solution viscosity obtained for the polybenzobisthiazole formation from a 12 wt % final polymer concentration in PPA.

The thermal and thermooxidative stability data for these polymers were obtained from TGA. The TGA data (10°C/min. heating rate) for 2,6-NaphPBT (entry 1, **Table 1**) are shown in **Figure 5** and **Table 2** describes the degradation behavior of all these polymers in inert atmosphere as well as in air.

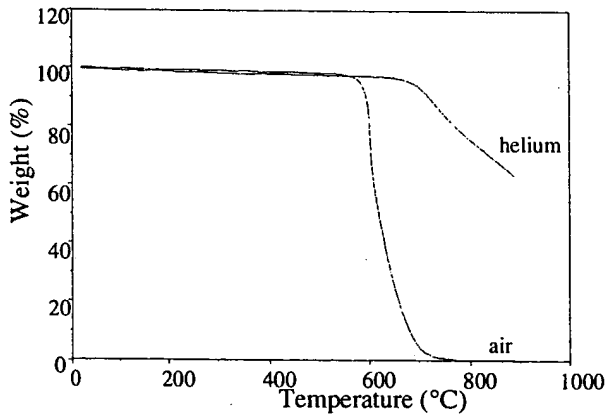


Figure 5. Thermogravimetric analysis of 2,6-NaphPBT in helium and in air

2,6-NaphPBT was found to be exceptionally stable with the onset of degradation occurring around 700°C in helium and 600°C in air.

Table 2. Thermal properties of Polybenzobisazoles incorporating Naphthalene-2,6- and 1,5-diyl units

Benzobisazole polymer	Degradation onset (He, °C)	Degradation onset (air, °C)
2,6-NaphPBT	692	595
2,6-NaphPBO	659	591
1,5-NaphPBT	610	548

Under the same conditions of analysis, the stability of 2,6-NaphPBO was nearly comparable to that of 2,6-NaphPBT. Though 1,5-NaphPBT polymer too exhibited high temperature stability, it had onset temperatures for degradation significantly lower than those of 2,6-NaphPBT.

Conclusions

Rigid-rod polybenzobisazoles incorporating naphthalene-2,6- and 1,5-diyl structural units were synthesized in high molecular weights. The lyotropic liquid crystalline behavior of the polymer dopes in PPA was demonstrated via the observation of optical birefringence. The polymers were found to exhibit high thermal and thermooxidative stabilities characteristic of aromatic heterocyclic rigid-chain polymers. Future studies will focus on the evaluation of the tensile as well as the compressive properties of spun fibers generated from these polymer systems.

Acknowledgement. The authors would like to acknowledge Marlene Houtz, University of Dayton Research Institute, Dayton, OH for the thermal analysis of the polymers reported in this paper.

References

(1) Wolfe, J. F.; Loo, B. H.; Arnold, F.E. *Macromolecules* **1981**, *14*, 915
(2) So, Y-H. *Prog. Polym. Sci.* **2000**, *25*, 137
(3) Matsuoka, T.; Kubota, F. *Jpn. Kokai. Tokkyo. Koho* **1998**, JP 10158213 (assigned to Toyobo Co., Ltd., Japan)
(4) Sadanobu, J.; Inata, H. Book of Abstracts, 211th ACS National Meeting, New Orleans, LA **1996**, POLY-256
(5) Park, S.Y.; Lee, J.W.; Venkatasubramanian, N.; Dang, T.D.; Arnold, F.E.; Farmer, B.L. *Polymer Preprints (American Chemical Society)*, **2002**, *43(1)*, xxxx
(6) Wolfe, J.F. *Encyclopedia of Polymer Science and Engineering* **1988**, *11*, 601